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APPLICATION OF A RAPID QUENCH THERMOMAGNETIC ANALYZER TO AUSTEMPERED DUCTILE IRON

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13. ABSTRACT (Maximum 200 words) A thermomagnetic analyzer was fabricated to provide in-situ monitoring of isothermal decompositions of austenite in steels and related alloys. The analyzer provides a convenient and rapid means for establishing time-temperature-transformation characteristics of a ferrous alloy by cycling a given specimen through a series of thermal treatments and monitoring the transformations magnetically. The present report describes the apparatus and gives results on the transformation characteristics of austempered ductile iron (ADI). This analyzer was also used to investigate alternative processing procedures; the results indicate that an improved heat treatment is available for large ADI components.				
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TABLE OF CONTENTS

ACKNOWLEDGEMENTS	ii
INTRODUCTION	1
EXPERIMENTAL	2
RESULTS AND DISCUSSION	4
SUMMARY	5
REFERENCES	6

Tables

1. Composition of Austempered Ductile Iron Specimens	3
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List of Illustrations

1. Schematic of the rapid cool thermomagnetic analyzer	7
2. Photograph of the rapid cool thermomagnetic analyzer	8
3. Diagram of the steel quench block showing configuration of radial hole for thermocouple insertion and axial opening for specimen	9
4. Thermomagnetic analyzer output during austempering of ADI at 316°C for austenitizing temperatures of 900 and 980°C	10
5. Computer model calculations of cooling path for a 2-inch outside diameter rod for 200 and 400°C austempering molten salt baths	11
6. Thermomagnetic analyzer output for an ADI specimen that was first cooled to 175°C and held for 15 minutes at this temperature prior to austempering at 316°C	12

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INTRODUCTION

This report describes a thermomagnetic analyzer designed for monitoring isothermal decompositions of austenite in steels after rapid cooling from austenitization temperatures. The analyzer determines the time-temperature-transformation characteristics of an alloy by subjecting a given specimen to a series of thermal cycles and magnetically monitoring the transformations as they occur. The apparatus is particularly useful in monitoring effects of alloy composition variations so that heat treatments can be optimized. This procedure has clear advantages in convenience and time over the traditional metallographical methods of thermal processing, cutting, mounting, and determining progress of transformations by microscopy.

This report also describes results of the application of the analyzer to determine the transformation characteristics of austempered ductile iron (ADI). Currently, there is great interest in advancing the use of ADI, and a wide variety of compositions are under investigation in an attempt to enhance properties.

The analyzer is well-suited to assist in such efforts because (1) transformation properties are dependent on composition; (2) ADI has "low hardenability" necessitating a thermal analyzer that can quickly cool the specimen from its austenitizing temperature; and (3) austempering is an isothermal process.

For proper ADI processing, it is essential to establish the transition time for the end of the first stage (and initiation of the second) for a given austempering temperature since austempering for this length of time yields optimum mechanical properties. The data from this apparatus clearly show the characteristic two-stage feature of the ADI austempering transformations which allows measurement of this transition time.

At present, there is particular interest in applying ADI technology to larger components in an attempt to expand the use of ADI material. The basic limitation on component size is the limited cooling power of hot austempering baths which are usually operated in the 300 to 400°C range. Deleterious pearlite forms above 500°C, so the cooling rate in this range should be maintained as high as possible. Calculations of cooling paths for different austempering bath temperatures were conducted for large components. The results show that significant increases in cooling rates can be achieved in the vicinity of the pearlite knee with proper selection of cooling paths. Experiments on the alternative processing procedure suggested by these results were conducted with this apparatus. The results indicate that an improved processing method is available for larger ADI components.

EXPERIMENTAL

Figure 1 is a schematic of the rapid cool thermomagnetic analyzer and Figure 2 is a photograph of the apparatus. The apparatus consists of a vertical arrangement of an austenitizing furnace at the top and an austempering furnace at the bottom so that the specimen can be moved from one to the other by raising or lowering using a fine thermocouple wire. This apparatus is similar in concept to a system designed by Bapu, Bhat, Parker, and Zackay at the University of California at Berkeley (ref 1). The present system is simpler in design, fabrication, and operation; it uses "off-the shelf" items for most of the main components, and the data acquisition is computerized.

The progress of the isothermal transformation from non-ferromagnetic austenite to the various ferromagnetic decomposition products, such as bainite, is monitored magnetically with a set of coils that surround the specimen and the narrow austempering furnace. The coil set consists of a primary coil that magnetizes the specimen with an alternating field and a secondary coil that detects the extent of magnetization of the specimen. This apparatus uses a commercial system, Magnatest-S, from Forster Instruments (Pittsburgh, PA) for the coil set and analysis instrumentation. The output is registered digitally as real and imaginary components of the secondary coil impedance. The dominant contribution is the imaginary component from the coil inductance. In the present setup, the analysis instrumentation is interfaced to a computer for data recording and control of the experiment.

A narrow (0.75-inch diameter) austempering furnace is used to maximize magnetic coupling between the coils and the specimen. The furnace is vacuum jacketed for maximum thermal isolation to avoid undue heating of the close fitting coil arrangement. The furnace windings are bifilarly wound to preclude spurious magnetic fields in the pickup coil. This specialized furnace was obtained from Princeton Applied Research (Princeton, NJ).

In order that magnetic response (coil/specimen inductance) be approximately proportional to volume transformed, it is important that the specimen be fabricated in rod form with a proper length-to-diameter ratio. The appropriate configuration for our purposes is a cylinder 1/8 inch in diameter and 3/4 inch in length.

Because specimens are thin, it is essential that the surface be protected from degradation by oxidation and decarburization at high temperatures. This is accomplished by electroplating the specimens with copper and providing a protective atmosphere of 90 percent helium and 10 percent hydrogen during thermal treatments.

The Berkeley apparatus uses a circulating hot liquid (molten salt) as the medium into which the specimen is rapidly immersed to achieve rapid cooling from the austenitizing temperature (cooling times are five seconds or less) and the long term isothermal hold. The difficulties associated with circulating molten salts are circumvented in the present apparatus by the use of a "quench block" shown in Figure 3. The quench block is a steel cylinder located between the austenitizing furnace and low temperature austempering furnace. It is maintained at approximately room temperature by close contact with the large brass coupling between the quartz tube and lower furnace assembly. The cylinder has a radial and an axial hole that permit simultaneous insertion of the specimen from above and insertion of a thermocouple wire through

the side. The thermocouple wire serves the dual role of dynamically monitoring the specimen temperature as it rapidly cools and pinning the specimen in place within the cylinder. Thus, the specimen can be released at the instant the target temperature is reached by simply retracting the thermocouple wire and permitting the specimen to fall into the isothermal furnace. In the present arrangement, the thermocouple wire is inserted into the side of the quench block through the opening in the brass coupling that also serves as the inlet port for the protective gas.

If the specimen were permitted to drop directly into the lower furnace from the austenitizing furnace without the quench block, the specimen temperature would approach the target temperature asymptotically, requiring several minutes to reach to within two or three degrees of the target temperature. This is too slow, because in many cases high temperature products can nucleate and grow before the target temperature is reached. Tests with the quench block arrangement show that the specimen temperature reaches the target temperature in ten seconds or less, which is sufficient for most practical applications.

A calibration procedure is required for proper operation of the quench block because of the small additional cooling of the specimen ($\sim 20^{\circ}\text{C}$) between the block and the low temperature furnace. The calibration procedure requires a well-annealed standard specimen fabricated from a ferromagnetic metal or alloy that has a Curie temperature near the upper end of the range of the isothermal temperatures of the experiments. For our measurements in the 300 to 400°C range, nickel and/or nickel-cobalt alloys are appropriate because the high sensitivity of the magnetization to temperature in this range permits the standard specimen's magnetization to serve as accurate measures of the specimen's true temperature. This temperature can be recorded within seconds of the release of the standard specimen into the low temperature furnace. Thus, by performing a series of magnetization measurements of the temperature lag of the standard for a range of thermocouple readings, a calibration plot can be established for application to other specimens of similar geometry.

As a check on the present design, a Princeton Applied Research vibrating sample magnetometer was used to determine the percent transformation of austenite after the austempering process in several specimens. This is a standard technique for retained austenite determination employing a saturation magnetic field. The results indicate that with the present arrangement, the Magnatest-S output varies linearly with volume transformed to within approximately ± 3 percent, which is a very desirable feature for quantitative analyses of transformation processes.

Table 1 lists the composition of the ADI specimens used in the present experiments.

Table 1. Composition of Austempered Ductile Iron Specimens

C	Si	Mn	Ni	Cu	Mo	P	S
3.7	2.5	0.3	1.0	0.9	0.0	0.01	0.01

RESULTS AND DISCUSSION

Figure 4 shows the austempering transformations at 316°C following a rapid cooldown from austenitizing temperatures of 900 and 980°C. The large differences in transformation rates and in the amount of untransformed austenite remaining at the end of the process are due to the difference in the amount of carbon that dissolves in austenite at these two austenitizing temperatures.

Also shown in Figure 4 is the transformation at 400°C following austenitization at 900°C; the two-stage nature of the transformation is clearly exhibited here. The initial transformation is the formation of fine-grained acicular ferrite which, in combination with the untransformed austenite, is considered responsible for the high strength and toughness of ADI. The second transformation beginning at approximately 200 minutes is the decomposition of the untransformed austenite to ferrite and carbides. The presence of any appreciable quantity of this second transformation product is known to yield inferior mechanical properties.

The toughness properties of ADI are degraded by the formation of pearlite when the cooling rate is too slow. Figure 5 compares the calculated temperature-time path for the center of a long 4-inch diameter solid cylinder after transfer from austenitizing to austempering baths at two different temperatures. The austenitizing bath temperature is 900°C, while the two austempering temperatures are 400 and 200°C, respectively. The significant point is that the cooling rate for the 200°C bath in the 500 to 600°C range is roughly a factor of two faster than for the 400°C bath. If the component can be cooled initially to a low temperature just above M_s where the transformation rate is essentially zero, and subsequently heated to the desired austempering temperature from below, then one can take advantage of the resulting faster cooling rate in the pearlite zone to heat treat larger components.

Figure 6 is the output from an experiment where an austenitized ADI specimen was first cooled to 175°C and then heated to 316°C austempering temperature from below; the austenitization temperature in this case was 980°C. The objective here was to simulate our proposed heat treat procedure where large components are first quenched into a relatively cool bath (above M_s) to enhance the cooling rate in the critical region of the pearlite knee. The low M_s temperature ($< 170^\circ\text{C}$) for these compositions allows such a procedure. As Figure 6 shows, there is no detectable bainite transformation in this temperature range (just above M_s), so the component may be held at this temperature until uniform component temperature is achieved.

There is often a small amount of transformation to bainite during the heatup phase to the austempering temperature as in this case. This transformation on heatup should not be deleterious to properties, since the transformation products are a small fraction of the whole and are all ferritic/bainitic in this range.

SUMMARY

The design and fabrication of a new magnetic thermal analyzer was described. The capabilities of the apparatus were demonstrated with measurements on ADI material. The analyzer was able to establish all the characteristic features of the ADI transformations from austenite to ferrite in a timely and convenient manner that avoids the specimen preparation difficulties of standard microscopy methods.

Experiments were also conducted on an alternative heat treatment procedure for large components. In this proposed method, the component is initially immersed into a relatively low temperature bath prior to the austempering procedure to effect more rapid cooling in the vicinity of the pearlite knee. The data suggest that a cooling rate of roughly a factor of two faster than in the higher temperature bath can be achieved, thereby permitting substantially larger ADI components to be successfully heat treated. The data from the thermomagnetic analyzer confirm that the proper transformation products are produced by this suggested process.

These results illustrate the convenience and commercial potential for application of this analyzer for monitoring and characterizing transformations in a broad range of ferrous alloy systems.

REFERENCES

1. B.N.P. Bapu, M.S. Bhat, E.R. Parker, and V.F. Zackay, *Met. Trans. A*, Vol. 7A, 1976, p. 17.

Thermomagnetic Analyzer

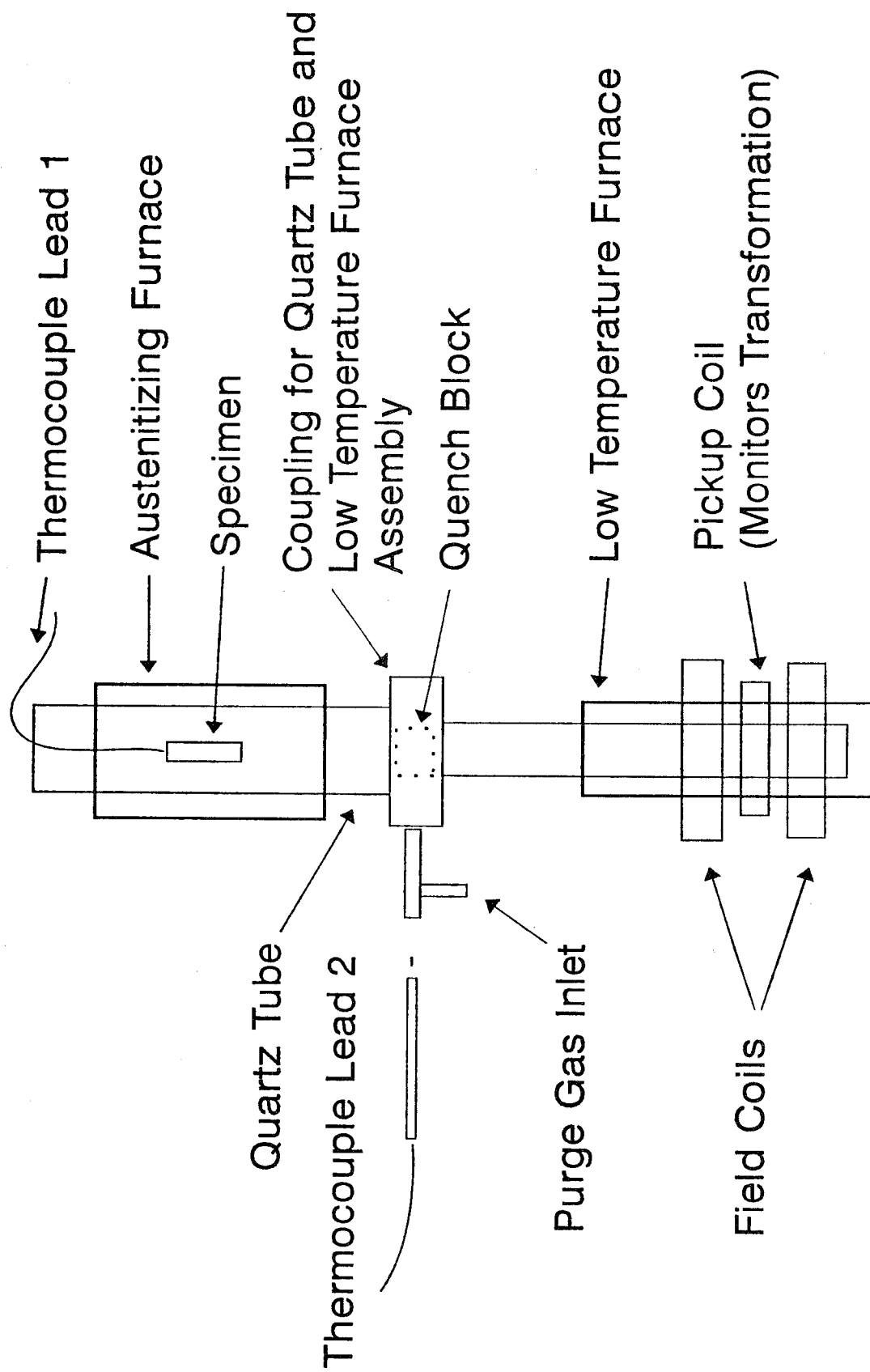


Figure 1. Schematic of the rapid cool thermomagnetic analyzer.

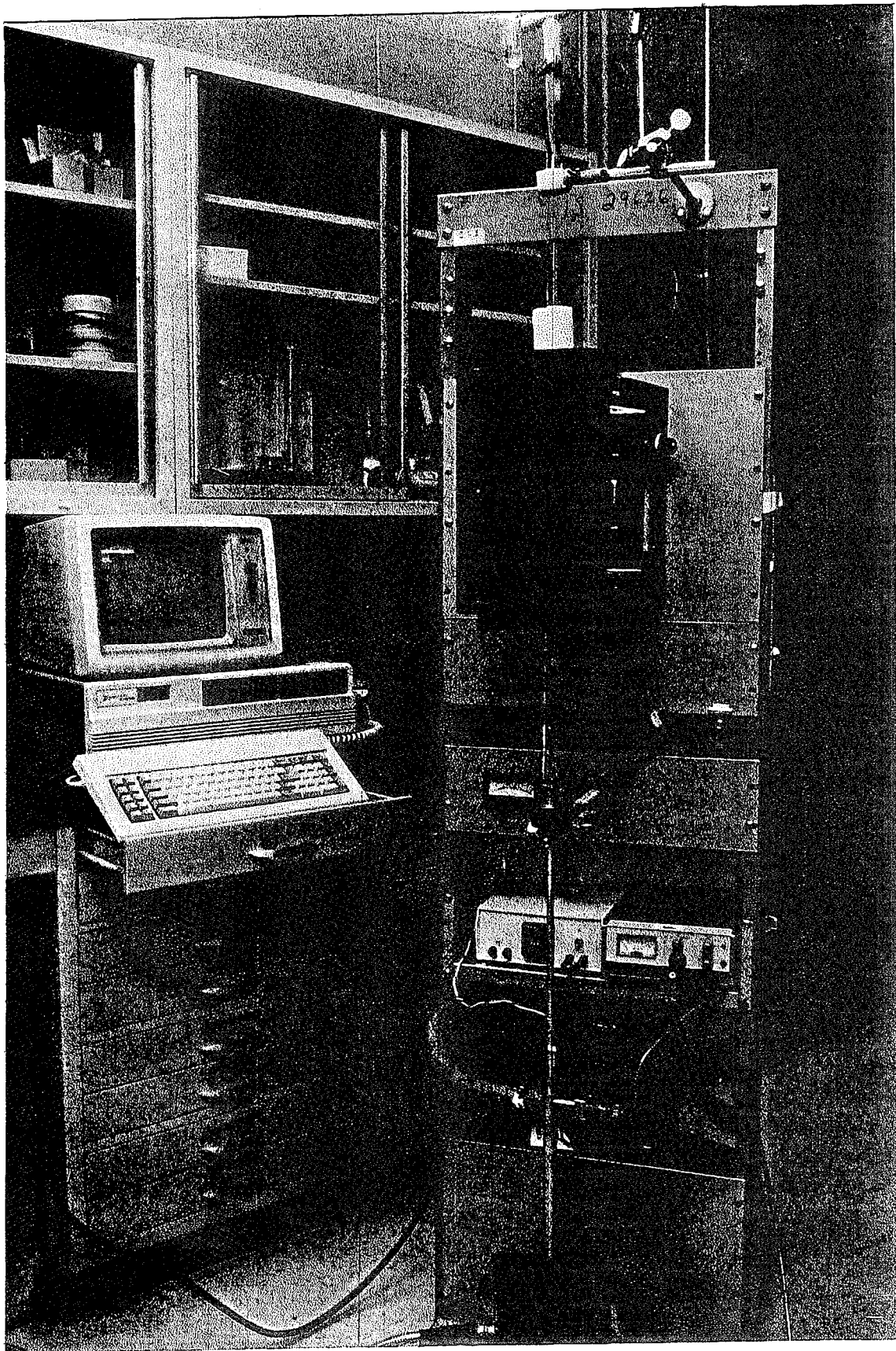


Figure 2. Photograph of the rapid cool thermomagnetic analyzer.

Quench Block Assembly

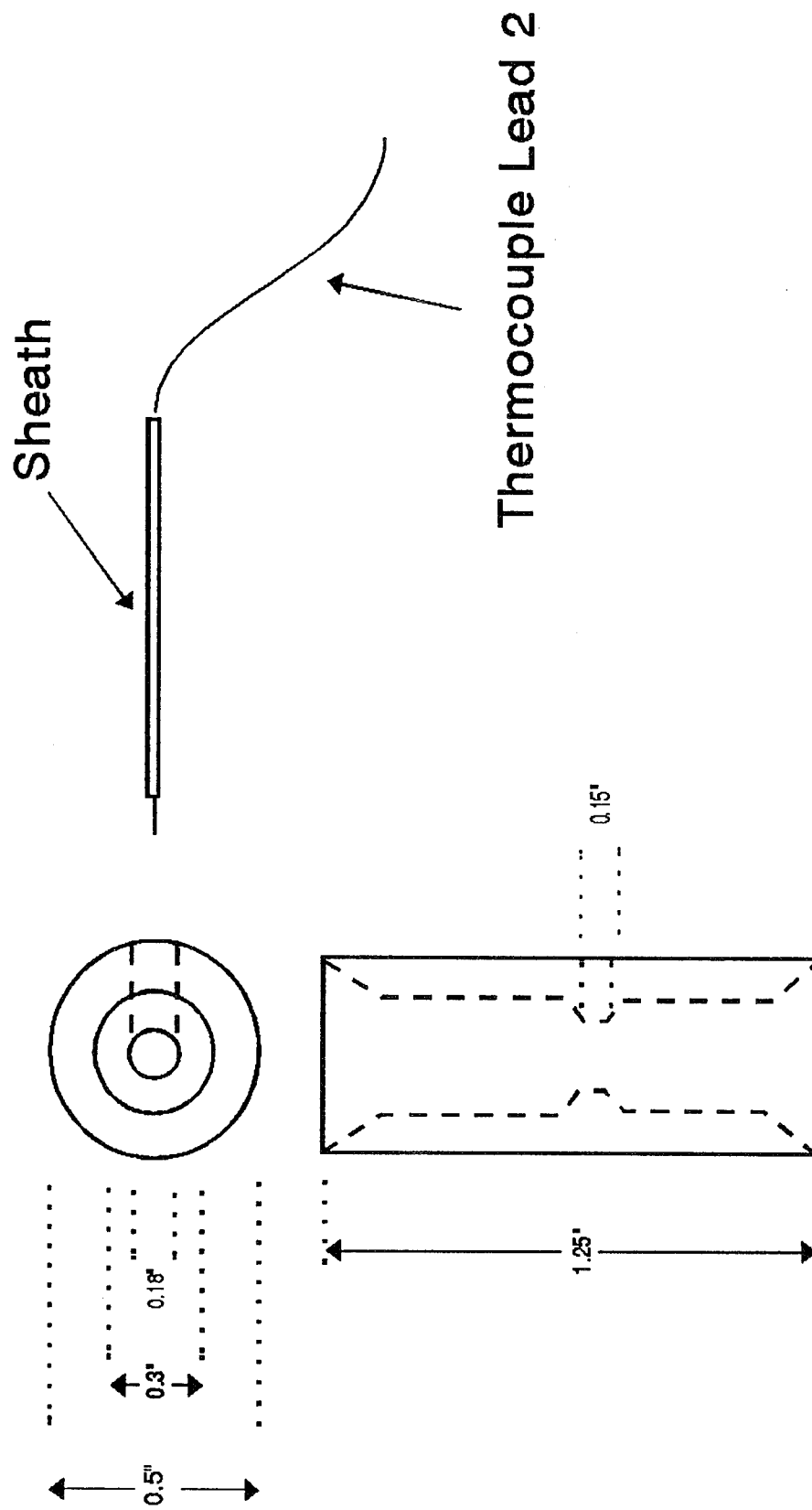


Figure 3. Diagram of the steel quench block showing configuration of radial hole for thermocouple insertion and axial opening for specimen.

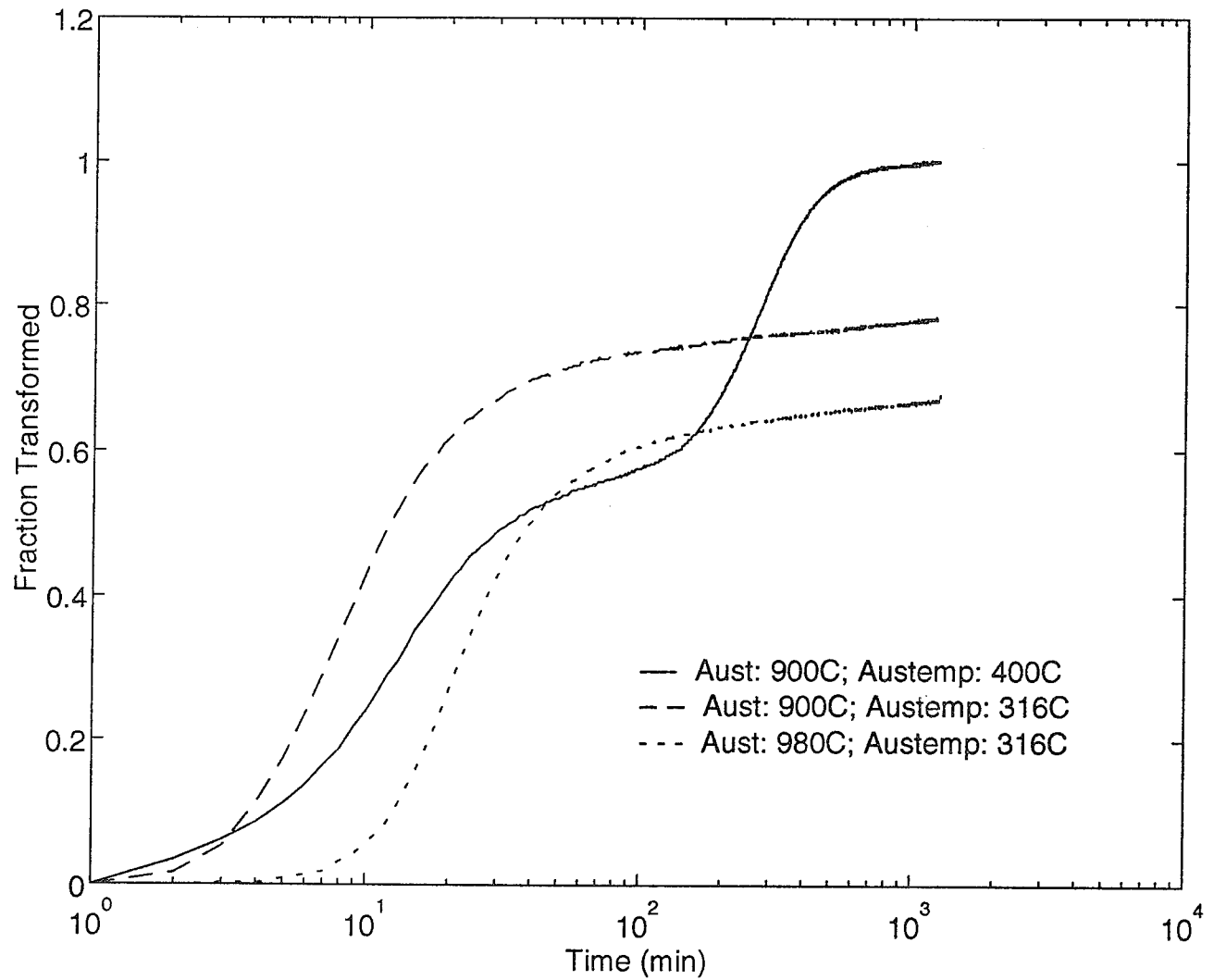


Figure 4. Thermomagnetic analyzer output during austempering of ADI at 316°C for austenitizing temperatures of 900 and 980°C. Also shown is the two stage nature of the austempering transformation at 400°C after austenitizing at 900°C.

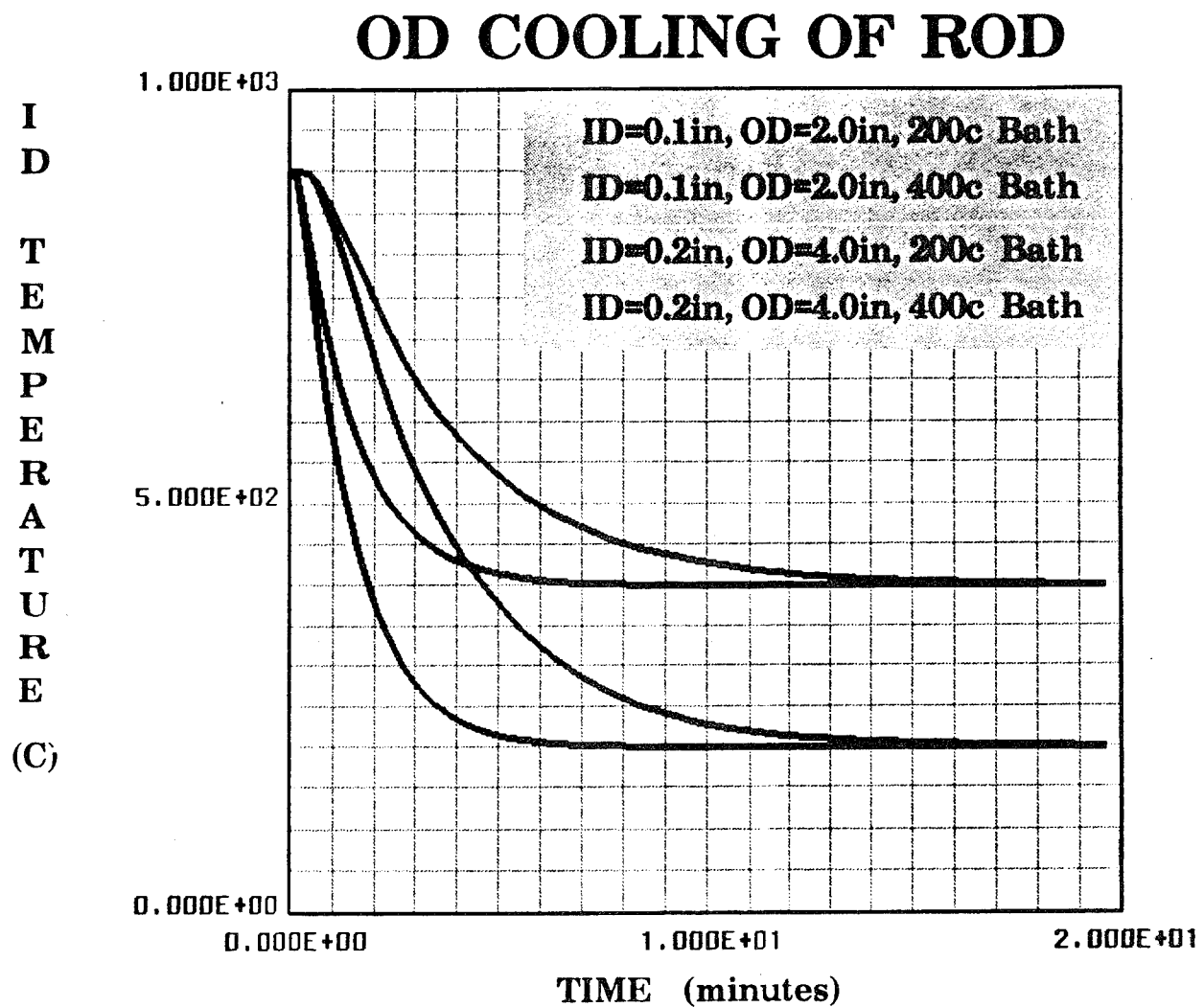


Figure 5. Computer model calculations of cooling path for a 2-inch outside diameter rod for 200 and 400°C austempering molten salt baths. The cooling rate in the 500 to 600°C pearlite range is roughly a factor of two faster for the 200°C bath.

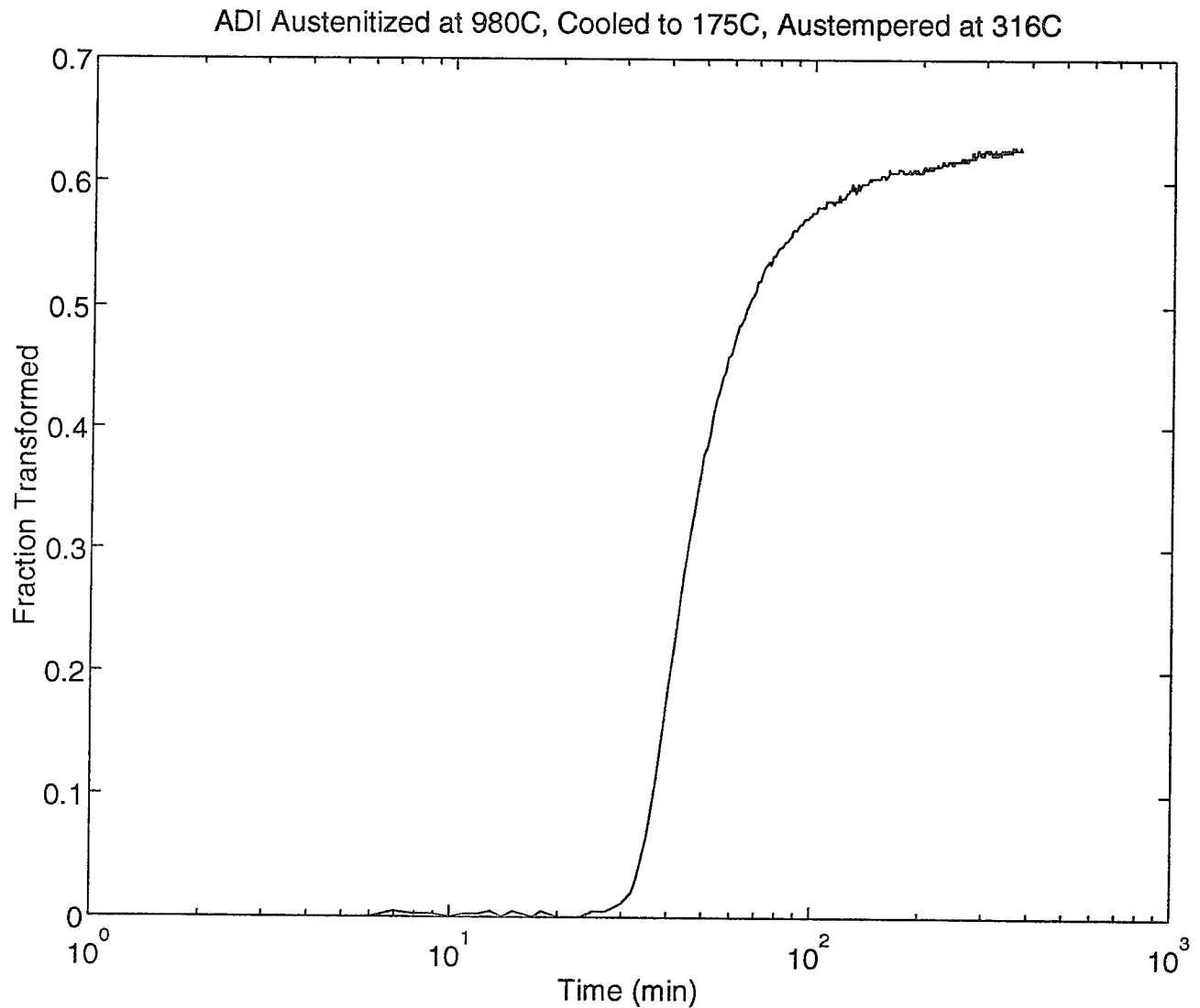


Figure 6. Thermomagnetic analyzer output for an ADI specimen that was first cooled to 175°C and held for 15 minutes at this temperature (where the transformation rate is essentially zero) prior to austempering at 316°C. The austenitization temperature was 980°C. The results demonstrate the viability of the proposed two-stage process for heat treating large components.

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